Canine accelerant detectors and problems with carpet pyrolysis products

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Dogs used as detectors for accelerants in controlled tests and actual fire scenes sometimes respond positively to burnt carpets and rubber underlays on which no flammable/combustible liquid accelerant has been placed. This paper reports on the identification of carpet and underlay pyrolysis products to which canines had responded falsely positive during validation and certification testing. The samples of burnt carpet and underlay were examined for pyrolysis product profiles using passive headspace concentration/solvent elution and analysis by capillary column gas chromatography. The results were compared to pyrolysis products prepared in the laboratory by Curie-point and tube pyrolysis from separate components of the carpets and underlays. The carpet pile, adhesive, plastic mesh and rubber polymeric compounds were identified as nylon 6/6, styrene-butadiene, ethylene-vinylacetate-indene, polypropylene, styrene-butadiene-isoprene and poly(1-butene)-polyethylene. It is clear that careful laboratory analysis of fire debris is required to confirm the presence of specific volatile accelerants.

Les chiens utilisés comme détecteurs de produits accélérateurs de feu, dans des tests contrôlés ou sur les lieux de feux réels, répondent parfois positivement à des tapis brûlés et de caoutchouc sur lesquels aucun liquide inflammable ou combustible n'avait été placé. Cet article décrit l'identification des produits de pyrolyse provenant de tapis et de sous-couches de caoutchouc auxquels les chiens avaient répondu positivement ou à tort durant les tests de validation et de certification. Les échantillons de tapis brûlés et de sous-couches ont été examinés pour leurs profils de produits de pyrolyse, en utilisant une méthode de concentration par espace de tête, élution par solvant et analyse en chromatographie en phase gazeuse sur colonne capillaire. Les résultats ont été comparés avec des produits de pyrolyse, préparés dans les laboratoires par point de Curie et pyrolyse en tube et des composés séparés des tapis et des sous-couches. Les produits des tapis, les colles, l'émail plastic et les composés polymériques du caoutchouc ont été identifiés comme du nylon 6/6, du styrène-butadiene, de l'éthylène vinylacétate-indène, du propylène, du styrène-butadiene-isoprène et du poly(1-butène)-polyéthylène. Il est clair que l'analyse soigneuse en laboratoire des débris provenant du feu est nécessaire pour confirmer la présence d'accélérants volatiles spécifiques.

Hunde, die zur Detektion von Brandbeschleunigern eingesetzt werden, reagieren sowohl in kontrollierten Tests als auch in tatsächlichen Brandlegungsfällen manchmal positiv auf brandbetroffenes Teppichbodenmaterial und deren Rückenbeschichtung, obwohl kein Brandbeschleuniger verwendet worden ist. Es wird über die Identifizierung der Pyrolyseprodukte von Teppichbodenmaterialien und deren Rückenbeschichtungen berichtet, auf welche die Hunde bei der Feststellung ihrer Detektionsleistung falsch positiv reagiert haben. Die Pyrolyseprodukte brandbeaufschlagter Teppichebodenbeläge und Rückenbeschichtungen wurden mittels der passiven Dampfraumanreicherung bzw. der Flüssigelution angereichert und gaschromatographisch analysiert. Die Ergebnisse wurden verglichen mit den Pyrolyseprodukten der Einzelkomponenten des Teppichmaterials und der Rückenbeschichtung, welche im Labor durch Curiepunkt-Pyrolyse bzw. die übliche Ofenpyrolyse gewonnen worden sind. Der Teppichflor, die Kleber, das Trägergewebe und die Rückenbeschichtung sind als Nylon 6.6, Styrenbutadien, Ethylenvinylacetatinden, Polypropylen, Styrenbutadienisopren und Poly-(1-Buten)-Polyethylen identifiziert worden. Es wird deutlich, daß sorgfältige Laboranalysen erforderlich sind, um in Brandrückständen insbesondere flüchtige Brandbeschleuniger nachzuweisen.

Los perros usados como detectores de aceleradores en tests controlados y escenas de fuego reales, a veces responden positivamente a los alfombras y arpilleras de goma quemadas en las que no habían sido colocados ningún liquido acelerador inflamable o combustible. Este informe trata de la identificatión de productos de pirólisis de alfombras y esterillas a los que los perros han respondido produciendo falsos positivos durante la validación y los tests de certificatión. Las muestras de alfombra y esterilla quemados fueron analizadas en busca de perfiles de productos de pirólisis usando cromatografía de gases acoplada con analizador de espacio en cabeza. Los resultados fueron comparados con productos de priólisis preparados en el laboratorio por Curie-point y tubo de pirólisis de componentes separados de las alfombras y esterillas. Los adhesivos de alfombras, mallas plásticas y compuestos poliméricos de goma fueron identificados como nylon 6/6, estireno-butadieno, etileno-vinalaceteto-indano, polipropileno, estereno-butadieno-isopreno y poli (l-buteno) polietileno. Esta claro que se requiren cuidados análisis de laboratorio de los restos de incendios para confirmar la presencia de aceleradores volátiles específicos.

Key Words: Forensic science; Criminalistics; Arson investigation; Canine accelerant detectors; Carpet/rubber underlay pyrolysis products; Curie-point pyrolysis; Static headspace sampling; Gas chromatography; Mass spectrometry.

Introduction

Arson investigation has proved to be a great challenge to the fire scene investigator and the laboratory analyst alike and each is always willing to try new methods or techniques that may be of assistance. One area of mutual interest is the use of flammable liquid/vapour detectors or 'sniffers' at fire scenes. Electronic accelerant sniffers have been proved as a useful tool for fire scene investigation and are now in routine use in the United States of America and Australia. Canine accelerant detection teams (dog and handler) were first developed during the mid-1980s by the US Department of the Treasury (Bureau of Alcohol, Tobacco and Firearms) and the Connecticut State Police (CSP) [1,2]. Since 1986, when the first operational team was employed (CSP), many teams have been placed into operation with both public (government) and private (non-government) investigators in the United States. The success of such teams has been reported in very favourable terms [3]. To date only one dog is operational in Australia (in the State of New South Wales).

The State Farm Insurance and Aetna Insurance companies (USA) currently provide funding for the training of canine teams [4]. Some independent validation and certification [5,6] and evaluation [7,8] of canine teams has been carried out. This paper reports on the application of pyrolysis techniques [9] to the determination and identification of carpet and rubber pyrolysis products to which canines responded falsely positive in validation and certification tests [6], undertaken in California during 1992–1994. These showed the canines to be highly accurate (>80% accuracy) in detecting the presence of volatile organic compounds (flammable/combustible liquid accelerants or pyrolysis products) in burnt substrates, in agreement with previously published information [3,7,8]. However, four of the seven canines in the 1994 tests [6] responded falsely positive to a variety of burnt substrates. Results from these tests demonstrated the canines to have a false positive response rate of 26% (nine out of 35 tests), which was in agreement with mixed matrix discrimination tests in an evaluation of accelerant detection canines [8]. The 35 tests comprised 7 dogs x 5 blanks of burnt substrate. The nine false positive responses related to five samples of burnt carpet, one sample of burnt urethane foam/fabric, two samples of burnt plastic (polyethylene/polystyrene) and one sample of burnt plywood. A similar result, in which burnt carpet caused the most false positive responses, has also been reported in other canine tests [7].

TABLE 1 Constituents of carpet samples A13, D16, A5 and B19, used in the canine tests [6]

Carpet	*Pile/Fibre type*	*Base*
A13	Cut Pile/Synthetic fibre	Plastic Mesh/Adhesive/ Plastic Mesh
D16	Cut Pile/Synthetic fibre	Plastic Mesh/Adhesive/ Plastic Mesh
A5	Cut Pile/Synthetic fibre	Plastic Mesh/Adhesive/ Jute
	Rubber Underlay	Green Rubber
B19	Loop Pile/Synthetic Fibre	Adhesive/Black Rubber

Experimental

In all the tests undertaken in California [6] only four types of carpet were used, comprising a synthetic fibre pile and a backing matrix of plastic/ jute or rubber. The constituents of carpet samples A13, D16, A5, and B19, representative of these four types, are shown in Table 1.

Sample preparation and canine testing

A series of tests was carried out in California during 1992–1994 [6], comprising discrimination and scene examination. The discrimination tests involved the preparation of 20 targets, including five blanks, of various burned common substrates (carpet and rubber underlay, fabric and foam, plastic, plywood) for each canine team. Fifteen samples were spiked with 10–50 μl of various flammable liquid accelerants (acetone, lighter fluid, lacquer thinners, evaporated petrol, kerosene and diesel).

The targets were prepared in the laboratory and placed into metal cans. The sealed cans were taken to an unburned dwelling where the samples were removed and presented to the canine teams as blind samples. After the reactions of the dogs were videotaped, the samples were recovered for laboratory analysis (passive headspace-charcoal adsorption /solvent elution). Samples to which the dogs alerted were considered positives. Those positive samples which did not contain added (spiked) flammable liquids were considered false positives. These samples were selected for laboratory testing to confirm and identify the pyrolysis products that caused the canines to respond.

Carbon disulphide (spectrophotometric grade) was purchased from Fisher Scientific. Analytical grade n-pentane (Ajax Chemicals) was purified by refluxing with concentrated sulphuric acid (5% by volume) for 3 hours, followed by fractional distillation. Carbon strips were obtained from Albrayco Laboratories (Connecticut, USA), clean one litre metal paint cans from Mason/Sinclair Paint Company (Sacramento, USA), and Curie point pyrolysis wires and control unit from Horizon Instruments Ltd., (Heathfield, Sussex, England).

Gas chromatography-flame ionisation detection (GC/FID) was undertaken using Hewlett Packard 5890 model gas chromatographs, fitted with capillary columns, split/splitless injectors and flame ionisation detectors. A Shimadzu CR3A recorder or Hewlett Packard HP7475A plotter was used for data acquisition and production of chromatograms. Fused silica capillary columns and the operating parameters for the gas chromatographs are given in Table 2.

TABLE 2 Operating parameters for gas chromatographic techniques.

GC parameters	*carbon strip/solvent elution*	*Curie point pyrolysis/tube pyrolysis*
Column	Hewlett Packard HP1 capillary 0.25 mm i.d. x 15 m 0.25 µm thick bonded phase (methylsilicone)	J and W Scientific DB1 capillary 0.25 mm i.d. x 30 m 0.25 µm thick bonded phase (methylsilicone)
Carrier Gas	Helium 120 kPa head pressure 30 cm/min linear velocity	Helium 120 kPa head pressure 30 cm/min linear velocity
Injector	splitless mode, 250°C	splitless mode, 250°C
GC program		
initial temperature	60°C (1 min hold)	30°C (2 min hold)
ramp rate	30°C/min	8°C/min
final temperature	300°C	290°C
final hold	3 min	15 min
detector temperature	300°C	290°C

TABLE 3 Diagnostic pyrolysis products from in-house laboratory reference polymers and the literature [11–20].

Laboratory Reference Polymer	*Diagnostic Pyrolysis Products*	*No*	*Kovats Retention Index*
Styrene-	butadiene	1	480
Butadiene	toluene	2	756
Rubber	vinylcyclohexene	3	828
	ethylbenzene	4	852
	styrene	5	876
	vinyltoluene	6	967
Polypropylene	2,4-dimethyl-1-heptene (C9)	7	858
	2,4,6-trimethyl-1-nonene (C12) isomers	8	1050 and 1054
	2,4,6,8-tetramethyl-1-undecene (C15) isomers	9	1278,1284 and 1289
Polyisoprene	isoprene	10	500
Rubber	dimethylvinylcyclohexene	11	929
Malaysian natural rubber	dipentene	12	1000
Vinylacetate-	acetic acid	13	600
Indene	ethenyltoluene isomers	14	938,950,955
Hot melt adhesive	indene	15	997
Polyethylene	Homologous series of alkenes and alkanes including:		
	1,12-tridecadiene	16	1271
	1-tridecene	17	1281
	n-tridecane	18	1300
Poly(1-butene)	2,4-diethyl-1-octene (C12)	19	1082
(No 18, 139-0	2,4,6-triethyl-1-decene (C16) isomers	20	1377 and 1383
Density 0.91,	2,4,6,8-tetraethyl-1-dodecene (C20) isomers	21	1653,1667 and 1680
Aldrich and Co. USA)			

Gas chromatography-mass spectrometric detection (GC-MSD) was accomplished using a Hewlett Packard HP5890 GC, fitted with capillary column, split/splitless injector, coupled to a HP5970 mass selective detector. Mass spectrometer control, data acquisition and manipulation were carried out with a Hewlett Packard 5997C Chemstation. Mass spectral detection (EI) was carried out with 70 eV ionisation voltage (scan range 40–400 amu, scan cycle 1.2 seconds, threshold 500 counts). A fused silica capillary column (20 m by 0.25 mm id, 0.25–µm coating of polymethylsiloxane, Hewlett Packard HP 1) was used employing helium as the carrier gas (linear velocity 30 cm/minute). The inlet mode was splitless (purge off 25 seconds, inlet pressure 35 kPa) with an injection temperature of 250°C. The initial oven temperature of 30°C was held for two minutes, then programmed at 8°C/min to a final temperature of 290°C with a 15 minute hold time at that temperature.

Passive headspace-charcoal adsorption/solvent elution

The volatile hydrocarbons from the test samples were adsorbed onto a carbon strip by passive adsorption (sealed can containing the carbon strip and burnt substrate was heated in an oven for one hour at 65–70°C). The volatile organic compounds were desorbed from the carbon strip by elution with carbon disulphide (200–400 µl). An aliquot (2 µl) of the solvent was analysed by gas chromatography.

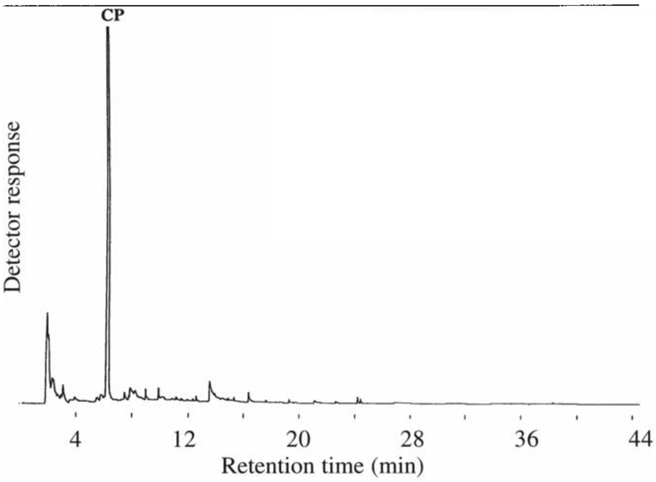

FIGURE 1 Pyrogram of fibres from carpet sample A13. CP = cyclopentanone

Curie point pyrolysis/gas chromatography (PY/GC)

To determine their polymer composition, a small sample (approximately 5mg) of the separate components (fibres adhesive, backing materials and rubber) of the carpets and underlays was placed into the hollow of a flattened, bent pyrolysis wire of Curie point 770°C. The pyrolyser interface temperature was held at 50°C.

The prepared wire was placed into a glass sample tube. The tube was inserted into the lower part of a metal probe which

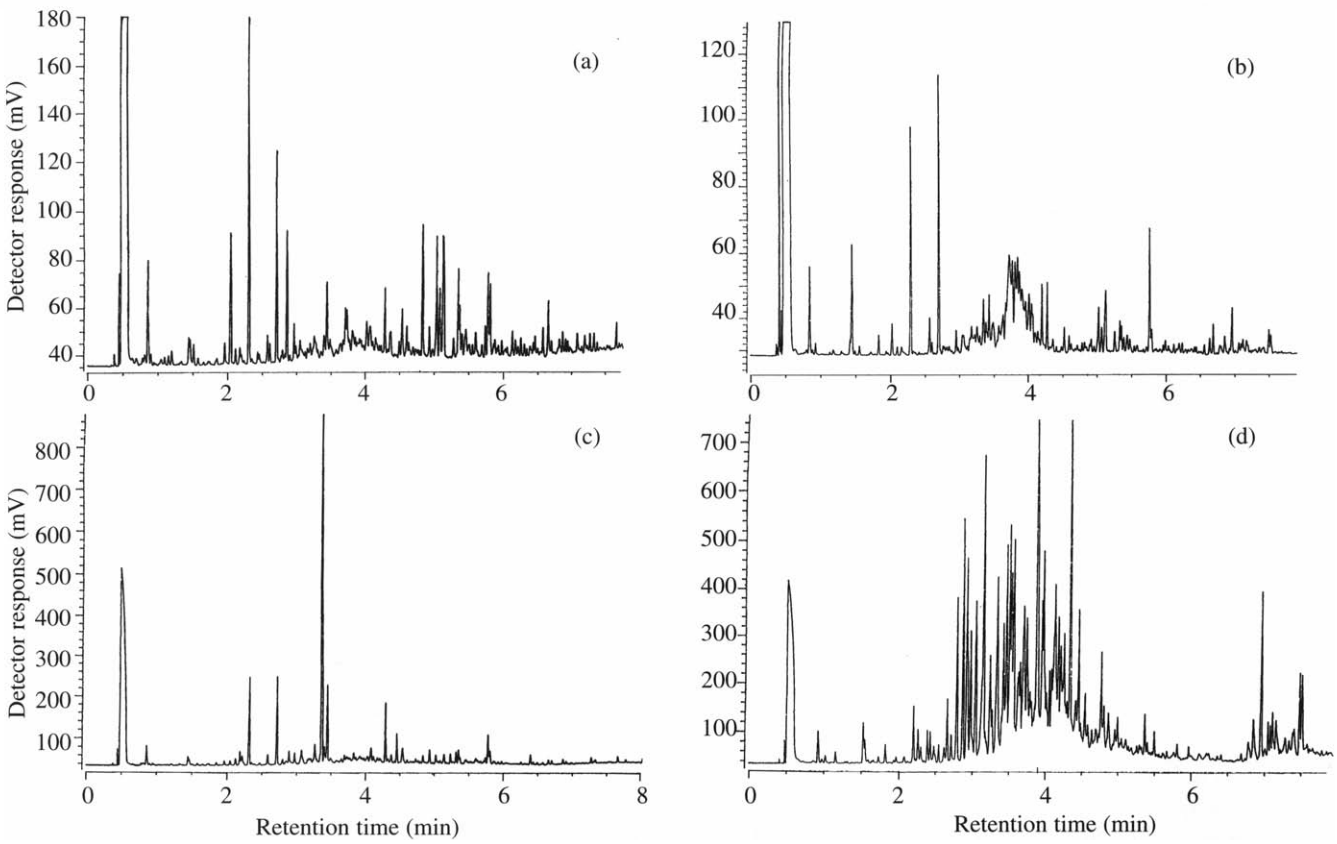

FIGURE 2 Chromatograms of pyrolysis products (carbon strip/CS2) from (a) burnt carpet A13; (b) burnt carpet D 16; (c) burnt carpet/rubber underlay A5; and (d) burnt carpet B19.

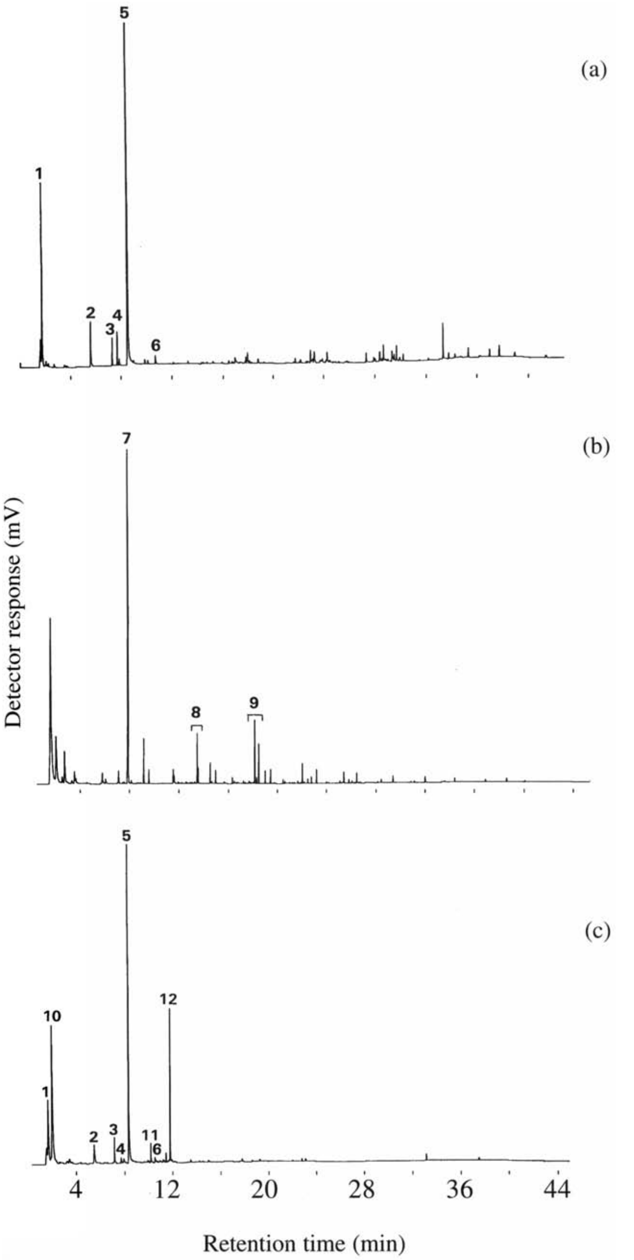

FIGURE 3 Pyrograms of (a) adhesive from carpet A13; (b) plastic mesh from carpet A13; and (c) rubber underlay from carpet A5. For explanation of numbered peaks, see Table 3.

was then placed in the pyrolyser, connected to the injection port of the gas chromatograph by a 0.5 mm i.d. stainless steel hypodermic needle [10]. The clamping nut and purge valve were tightened and the sample was pyrolysed (ramp time, less than 0.4 s to reach the pyrolysis temperature of 770°C) for 5 seconds and the pyrolysate flushed onto the column with helium (50 ml/min flow rate). The pyrolysis products were analysed by capillary column gas chromatography.

Tube pyrolysis/gas chromatography.
To determine their polymer composition, a small sample (approximately 5 mg) of the separate components of the carpets and underlays, as before, was placed in a 'u' shaped pasteur pipette, which had been sealed at one end. The tube was then sealed at the other end. The sample was pyrolysed by heating over a Bunsen flame until decomposition with the resultant pyrolysate condensing on the cooler areas of the tube. The sample was heated until char was formed. After allowing to cool to room temperature, the tube was broken open at one end and the pyrolysis products were removed by washing with pentane. The pentane (2 ml) was evaporated under nitrogen to a volume of approximately 200 µl. A 5 µl aliquot was analysed by capillary column gas chromatography.

The Curie point and tube pyrolysis techniques produced pyrolysis products which were compared to those of in-house laboratory reference polymers. Diagnostic pyrolysis products (numbered 1-21) from in-house laboratory reference polymers and the literature [11–20] are given in Table 3. Kovats retention indices [21] for the major diagnostic pyrolysis products are also included. The Kovats retention indices (polymethylsiloxane phase capillary column) were calculated using a diesel standard in pentane. The diesel comprised n-alkanes, octane to tricosane.

Results and discussion

The fibres of the carpet samples were identified by PY/GC as the polyamide, nylon 6/6. A pyrogram of fibres from carpet sample A13 (Figure 1) shows the major pyrolysis product, cyclopentanone [22].

Chromatograms of pyrolysis products (headspace/solvent elution) from the four different types of carpet that were used in the canine tests [6] are shown in Figure 2. The chromatograms for carpet samples A13 and D16 (Figures 2a and 2b) were similar to previously reported results [23–24] and, based on pattern recognition and Kovats retention indices, indicated pyrolysis products from styrene/butadiene and polypropylene. The chromatogram for carpet/underlay sample A5 (Figure 2c), indicated pyrolysis products from styrene/butadiene and isoprene. For carpet sample B19 (carpet with a rubber backing) the headspace volatiles chromatogram (Figure 2d) was complex, unusual, and not indicative of styrene-butadiene adhesives or styrene-butadiene-isoprene rubbers, which are often encountered in casework (unpublished observations). The chromatogram was similar to a mid-range petroleum distillate and the sample required further investigation involving tube pyrolysis/solvent extraction and analysis. Without further investigation (GC-MS), the chromatogram may be misinterpreted as volatiles from a flammable or combustible liquid.

Representative pyrograms of each polymer substrate for samples A13 (adhesive), D16 (plastic mesh) and A5 (rubber underlay), determined by Curie point PY/GC analysis, are shown in Figure 3. The numbers 1–21, used for peak identification in the chromatograms and pyrograms

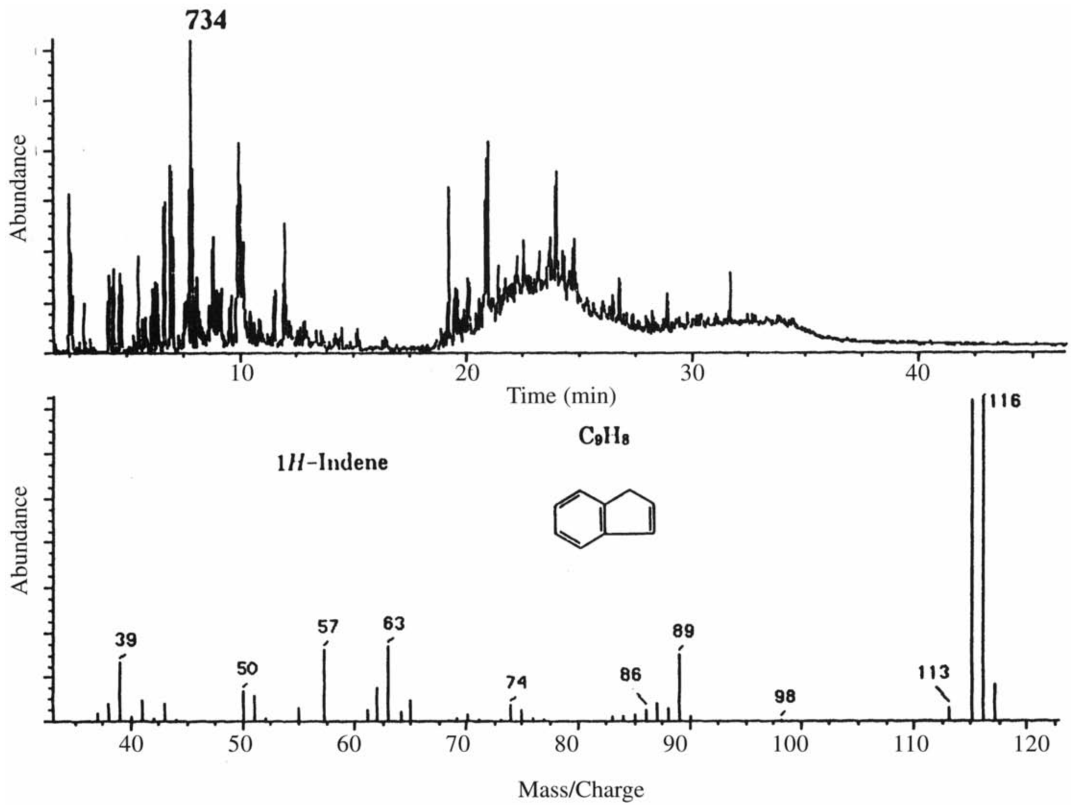

FIGURE 4 Chromatogram of tube pyrolysis products from burnt carpet B19.

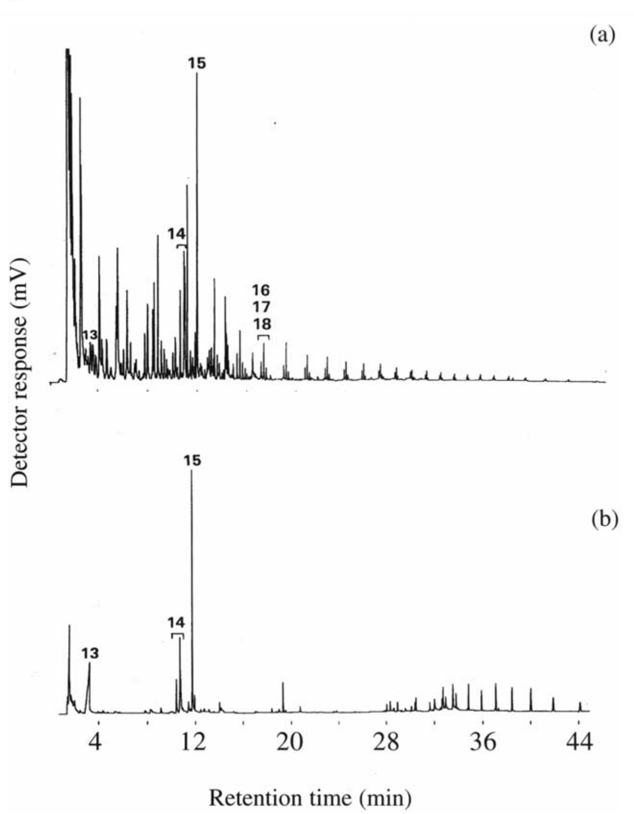

FIGURE 5 Pyrograms of (a) yellow adhesive from carpet B19 and (b) vinylacetate/indene adhesive. For explanation of numbered peaks, see Table 3.

(Figures 3–6), refer to diagnostic pyrolysis products listed in Table 3. These results confirm that the pyrolysis products from carpets A13, D16 and A5 were sourced from styrene-butadiene adhesive, polypropylene mesh and styrene-butadiene-isoprene rubber, respectively.

When a sample of carpet B19 was subjected to tube pyrolysis, and the pyrolysate was dissolved in pentane and analysed by GC-MS, the resultant chromatogram (Figure 4) showed a complex mixture of pyrolysis products, having a similar profile to that of volatiles collected from the canine test (cf Figure 2d). The major component was identified as 1H-Indene (scan 734) indicating the presence of an indene resin. Indene resins are widely used in adhesives, including hot melt formulations [25]. The components of the complex mixture which eluted after 19 minutes (Figure 4) were also present in a pentane extract of the unburnt carpet, which indicated that they were not pyrolysis products.

The GC-MS analysis of the pentane extract was negative for mineral oil or paraffin wax. Mineral oils and paraffin waxes are often recovered by solvent extraction from plastic mesh or jute backed carpets (unpublished observations). Mass spectra of components from the complex mixture did not match any in the MS reference library (NBS, Wiley). The compounds of the complex mixture were not examined further.

A subsample of yellow adhesive was removed from carpet B19, washed with pentane (to remove the background

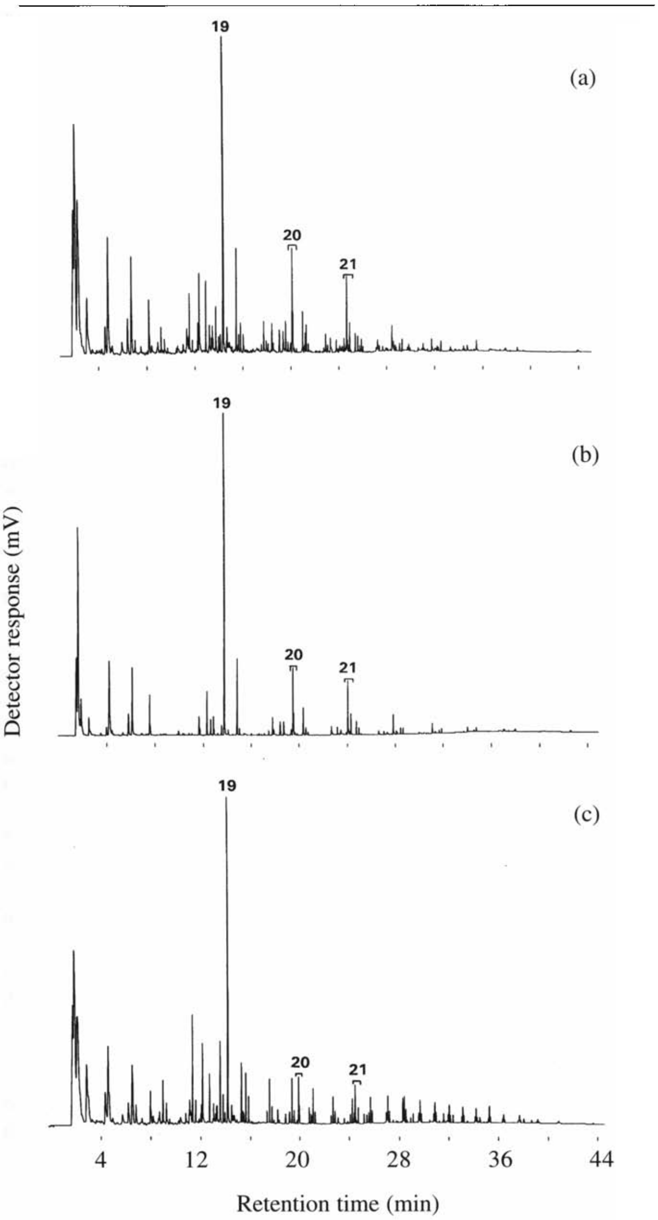

FIGURE 6 Pyrograms of (a) black rubber backing from carpet B19; (b) poly(1-butene); and (c) poly(1-butene)/polyethylene. For explanation of numbered peaks, see Table 3.

components), air dried and subjected to Curie-point PY/GC analysis. The resultant pyrogram (Figure 5a) showed indene, ethenyltoluenes and acetic acid, which are the major pyrolysis products of vinylacetate-indene adhesives. A pyrogram of a vinylacetate/indene hot melt adhesive reference polymer is shown for comparison (Figure 5b). The homologous series of triplets (clearly visible on the pyrogram in Figure 5a, eluting after 17 minutes) is indicative of polyethylene. The adhesive was identified as an ethylene/vinylacetate/indene copolymer. Ethylene/vinylacetate adhesives are used for carpet applications [26].

A subsample of the rubber backing from carpet B19, subjected to Curie-point PY/GC analysis, resulted in a pyrogram (Figure 6a) showing the major pyrolysis products to be 2,4-diethyl-1-octene, 2,4,6-triethyl-1-decene and 2,4,6,8-tetraethyl-1-dodecene, which are indicative of poly(1-butene). A pyrogram of poly(1-butene) reference polymer is shown for comparison (Figure 6b). The homologous series of triplets (noticeable in Figure 6a, eluting after 15 minutes) is indicative of polyethylene and is more clearly defined when a comparison is made with a pyrogram produced from the PY/GC analysis of a mixture of poly(1-butene) and polyethylene reference polymers (Figure 6c). The black rubber backing of carpet B19 was identified as a poly(1-butene)-polyethylene copolymer.

Conclusions

Canine accelerant detection team validation and certification tests [6] have shown that canines are suitable detectors for flammable/combustible liquid residues, although they may have difficulty in discriminating between flammable/combustible liquids and pyrolysis products from burnt carpets and rubber underlays. The importance of the test results should not be dismissed by the fire investigator. Carpet samples are collected more frequently from fire scenes than any other type of sample [27], and such samples will often contain complex mixtures of volatiles that the analyst may have difficulty in interpreting [28,29].

This paper demonstrates that canines can respond to pyrolysis products from polymers, such as nylon 6/6, styrene-butadiene, ethylene-vinylacetate-indene, polypropylene, styrene-butadiene-isoprene, and poly(1-butene)-polyethylene, sourced from carpet pile fibres, adhesives, plastic mesh backing material, rubber underlays and rubber backing materials.

For prosecution purposes it is advised that laboratory identification of flammable/combustible liquid residues or pyrolysis products from fire scene debris samples is paramount. This emphasises the need to verify all fire debris samples by gas chromatography with flame ionisation or mass spectrometric detection. Collection of unburned comparison samples (when available) is most useful in eliminating false indicators to materials normal to a firescene.

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